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Fatty Acid Based Ionic Liquids: A New Antistatic Agent For Floor Coating

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Abstract

This research was conducted to obtain cis-oleyl imidazolinium iodide ionic liquid as an antistatic agent in ceramic and wood floor coatings. Synthesis of cis-oleyl imidazolinium iodide ionic liquid was carried out by reacting diethylenetriamine and cis-oleic acid in a ratio of 1:2 which was reacted utilizing microwave irradiation followed by quaternary methylation using methyl iodide. Then, Fourier-transform infrared spectroscopy (FTIR), proton nuclear magnetic resonance (^{1}H NMR), and carbon-13 nuclear magnetic resonance (^{13}C NMR) instruments were used to characterize the synthesis results. The results showed that the synthesis of cis-oleyl imidazolinium iodide ionic liquid was successful. The electrostatic effectiveness of the cis-oleyl imidazolinium iodide mixed ionic liquid floor coating is statically dissipative because the surface resistance values are in the range of 106 – 109 Ω. Optimum point-to-point resistance in coating mixture is 108 Ω (2-9 wt%), ceramic floor coating 108 Ω (7-9 wt%), and wood floor coating 108 Ω (7-9 wt%). Meanwhile, the optimum ground resistance in the coating mixture is 108 Ω (2-9 wt%), ceramic coating 109 Ω (4-9 wt%), and wood floor coating 109 Ω (1-9 wt%). The coated floor has a yellow color due to iodide which makes it unsuitable for use on white surfaces. Dust adhesion test results show that the mixed-coated floor attracts much less dust than the non-ionic liquid coating.

Keywords: *cis-oleyl imidazolium, coating, floor, ionic liquid.*

1. Introduction

Research in the field of ionic liquids (ILs) is currently increasing as technology develops. ILS is a liquid salt that consists entirely of cations and anions and is a liquid at room temperature or at that temperature below 100°C. It should be noted that this temperature limit is not necessary for a substance to be considered an ILs [1]. ILs are distinguished from other salts, such as sodium chloride, which only become liquid at very high temperatures (>800°C) because of their tight structure and strong ionic interactions [2]. Then, the lower melting point of ILs is due to the increasing size and asymmetry of the constituent ions, so they can be paired together in different combinations to suit their thermophysical properties [3]. ILS has unique properties, such as very low volatility, wide liquid temperature range, good thermal stability, good dissolving ability, designable structure, high ionic conductivity, wide electrochemical window, and excellent microwave absorption ability [4]. Chemical, physical, and biological properties can be adjusted by the selection of different anions and cations. These distinctive, flexible, and unique characteristics of ILs are combined with the ability to function in various applications [5]. ILs are labeled as "solvents of the future", because they can be used in a variety of applications, particularly as a substitute for traditional solvents in industrial processes which are often toxic, flammable, and highly volatile. Its low vapor pressure leads to the assumption that ILs can also be considered environmentally friendly green solvents due to their low levels of atmospheric pollution, so it is important to note at this time that the manufacture, use, and disposal of the solvents themselves must also be considered [3]. In its application, ILs can be used as solvents or catalysts in synthesis processes, mobile phases for HPLC, columns in gas chromatography, biomass processing, or as solar panels, electrolytes in batteries, and fuel cells in electrochemistry [6]. Also, applications are found in industrial engineerings such as coatings, lubricants, and dispersing agents [7]. The properties of ILs as good conductors of electric current make them anti-electrostatic agents. Examples of applications to control the electrostatic state of clearcoats using special additives [8], also exist as core/skin composites used for antistatic coatings, and energy storage applications [9]. In this study, ILs were synthesized for antistatic purposes in floor coatings which were synthesized through one of the most frequently used coating methods, namely the UV-curing method. This method excels for its long durability, chemical resistance, stain resistance, and fast production. However, the weakness is that the polymer binder in this coating process is only an insulator and does not have anti-electrostatic properties. So that makes it very easy to experience static disturbances caused by electric charges due to contact or friction [10]. Interference from electric currents other than a direct danger to humans can cause damage to electronic goods, risk of explosion for flammable chemicals, or contamination due to the large amount of dust that sticks because they are attracted by static charges. For this reason, anti-electrostatic materials are needed to coat materials that can experience static disturbances such as on the floor surface. Based on this explanation, the IL is one option with many advantages over other alternatives. Imidazolium salt-based IL is reported to be used as an antistatic agent in wood floor coatings [10]. Based on the results of previous studies, ILs with imidazolium cations have high effectiveness and are very good as antistatic materials that have functional properties as metal-organic framework (MOF) materials and nano-micro media [11, 12]. Meanwhile, in other studies, it has been known that ILs containing anion I_2 have very high conductivity [13]. Here, the antistatic material developed in this research is an IL-based on fatty imidazolinium, which can be synthesized from fatty acids, as an antistatic agent in floor coatings. This allows for the synthesis of salt using local renewable sources such as palm oil and other vegetable oils. Based on the high resistance properties that cause a strong antistatic effect on alkyl imidazolium ILs and the presence of I^- anion support, the IL has high conductivity, so it is hoped that this IL will become an alternative antistatic material in the coating process with superior effectiveness than other materials. Then, the nature of the IL which is environmentally friendly and does not damage the appearance of the material [10], will be a profitable advantage for the industry.

2. Method

2.1. Materials and tools

The materials used in this study included: oleic acid-cis p.a 95% (Aldrich), methyl iodide p.a (Aldrich), diethylenetriamine p.a (Aldrich), technical ethyl acetate (Bratachem), technical methanol (Bratachem), floor tiles 30x30cm, 24x4cm teak wood, polyurethane coating for floors (AM 180 PU Coating) and wood (Propan PU Coating). The apparatuses used in this research include microwave LG30L 850W, glassware, a set of reflux apparatus, Buchner funnel, mercury thermometer, magnetic stirrer, electric heater, vacuum pump, a set of rotary evaporator, analytical balance, plastic wrap, aluminum foil, 100 mesh filter, Whatman 41 filter paper.

2.2. Synthesis method

Synthesis of cis-oleyl imidazolinium acetate IL was carried out by synthesizing cis-imidazoline, methylation-quaternary stage followed by anion replacement stage. The synthesis of cis-oleyl-imidazoline was carried out by heating with microwave irradiation [14], for the methylation-quaternary reaction step the reflux method was used [15]. The anion replacement reaction step is carried out by utilizing the principles of anion metathesis and Lewis acid-base [16]. The detailed steps are described below.

2.2.1. Synthesis of IL cis-oleyl imidazolinium (Step 1)

20 mmol (2.06 grams) of diethylenetriamine (DETA) and 40 mmol (11.29 grams) of fatty acids (cis-oleic acid) were put into a 100-mL pyrex beaker and stirred until evenly distributed. Then, the reagent mixture was irradiated using a microwave at 800W for 30 seconds and cooled to room temperature (25°C) in a fume hood. Then, the mixture was transferred to a three-necked round bottom flask and 80 mL of ethyl acetate was added and the mixture was heated to near the boiling point of ethyl acetate (40^0 C), for approximately 30 minutes. The mixture obtained was then filtered in a hot state using a Buchner funnel connected to a vacuum pump and continued with a little concentration using a rotary evaporator.

2.2.2. Synthesis of cis-oleyl imidazoline iodide through methylation-quaternization reaction (Step 2)

A total of 1 mole (13.65 g) of the cis-oleyl imidazolinium acetate IL that had been synthesized in the first stage was put into a three-neck round bottom flask that had been coated with aluminum foil and 2 moles (6.17 g) of methyl iodide were added to the flask. round base. The mixture was then refluxed at a constant temperature of 40^0 C while stirring using a magnetic stirrer for approximately 4 hours. Then, the results obtained were cooled to room temperature and concentrated using a rotary evaporator at a temperature of 80^0 C.

2.2.3. Chemical characterization

The instruments used to identify functional groups, study molecular structure and molecular interactions, and determine the arrangement (number) of carbon atoms in a molecule or IL compound, respectively, are Fourier-transform infrared spectroscopy (FTIR, SHIMADZU FTIR-8400), proton nuclear magnetic resonance (^{1}H NMR, JEOL JNM ECA-500 MHz, Japan), and carbon-13 nuclear magnetic resonance (^{13}C NMR, JEOL JNM ECA-500 MHz, Japan) and anion assay using $PbCl_2$. Then, to test the electrostatic activity, a surface resistance meter (Benetech Tester GM3110) was used. Detailed information for the interpretation of FTIR and NMR is reported elsewhere [17,18].

2.3. Ceramic and wood floor coating preparation

Cis-oleyl imidazolinium iodide is mixed into a coating agent (polyurethane) for ceramic and wood floors with a variation of 0-7 wt%. The mixture of coating materials is coated on the surface of the ceramic and wood floors in two layers.

2.4. Electrostatic activity test

Surface resistance tests, such as Point to Point Resistance and Ground Resistance are carried out on the coating material that has been made. Surface resistance tests and dust adhesion tests were also carried out on ceramics and wood that had been coated with the coating material, where the addition of cis-oleyl-Imidazolinium Iodide varied from 0-7 wt%.

3. Results and Discussion

3.1. Synthesis of IL cis-oleyl imidazolinium

The synthesis of Cis-Oleyl-Imidazolin was carried out using microwave irradiation. IL is synthesized from cis-oleic fatty acid and diethylenetriamine (DETA). Based on the materials used, the ILs are classified as fatty imidazolinium-based ILs. The synthesis method with irradiation was carried out because it has the advantage of reducing the use of solvents. After the synthesis process is complete, the yield of the IL produced is 68.4% with the physical state of the IL having a brownish yellow color and a characteristic odor. This yellow-brown color and characteristic odor indicate that the cis-oleyl imidazolinium product has been successfully formed. Figure 1 shows the steps in the synthesis of cis-oleyl imidazolinium.

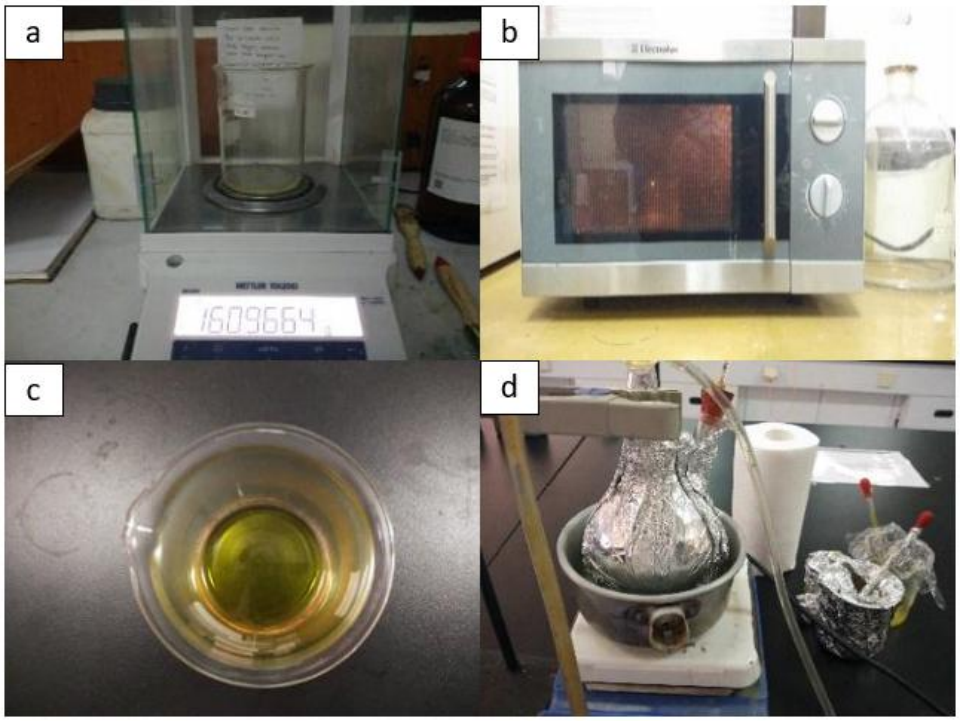

Figure 1. Stages of cis-oleic-imidazoline synthesis (a) weighing of oleic acid; (b) irradiation with microwaves; (c) irradiation results; (d) reflux with ethyl acetate solvent.

3.2. Synthesis of cis-oleyl-imidazolinium iodide

The synthesis of cis-oleyl-imidazolinium iodide was carried out by the methylation-quaternization reaction method. The cis-oleyl-imidazoline IL which had been successfully synthesized in the first stage was reacted with methyl iodide

by refluxing for 4 hours with the help of stirring by a magnetic stirrer. Methyl iodide is used because it is a good quaternary agent due to the large difference in electronegativity between methyl and iodide. To determine the success of the reaction, cis-oleyl-imidazolinium iodide was reacted with $PbCl_2$. The color change of $PbCl_2$ to yellow when reacted with IL indicates the presence of iodide and the quaternization reaction has been successful. Figure 2 shows the resulting color change when the cis-oleyl imidazolinium iodide IL is reacted with $PbCl_2$.

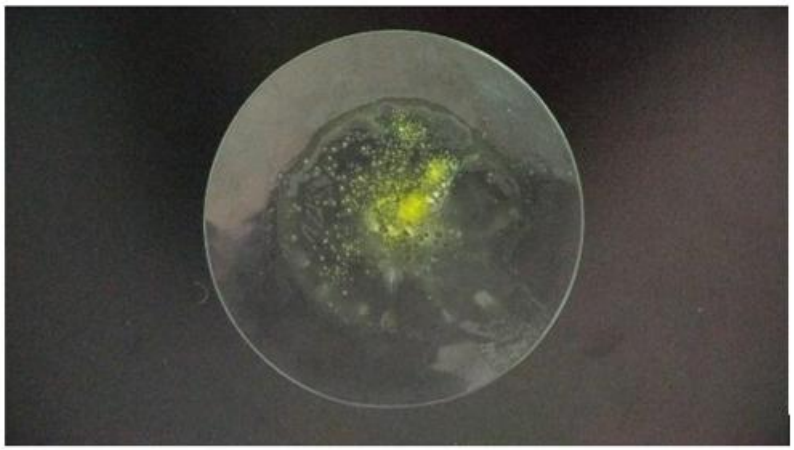

Figure 2. Color change results when cis-oleyl imidazolinium iodide IL is reacted with $PbCl_2$ Color change results when cis-oleyl imidazolinium iodide IL is reacted with $PbCl_2$

3.3. Chemical characterization of cis-oleyl-imidazolinium iodide by FTIR

Figure 3 shows the results of the identification of functional groups in the cis-oleyl-imidazolinium iodide IL using FTIR. Then, the functional groups available in Figure 3 are summarized in Table 1.

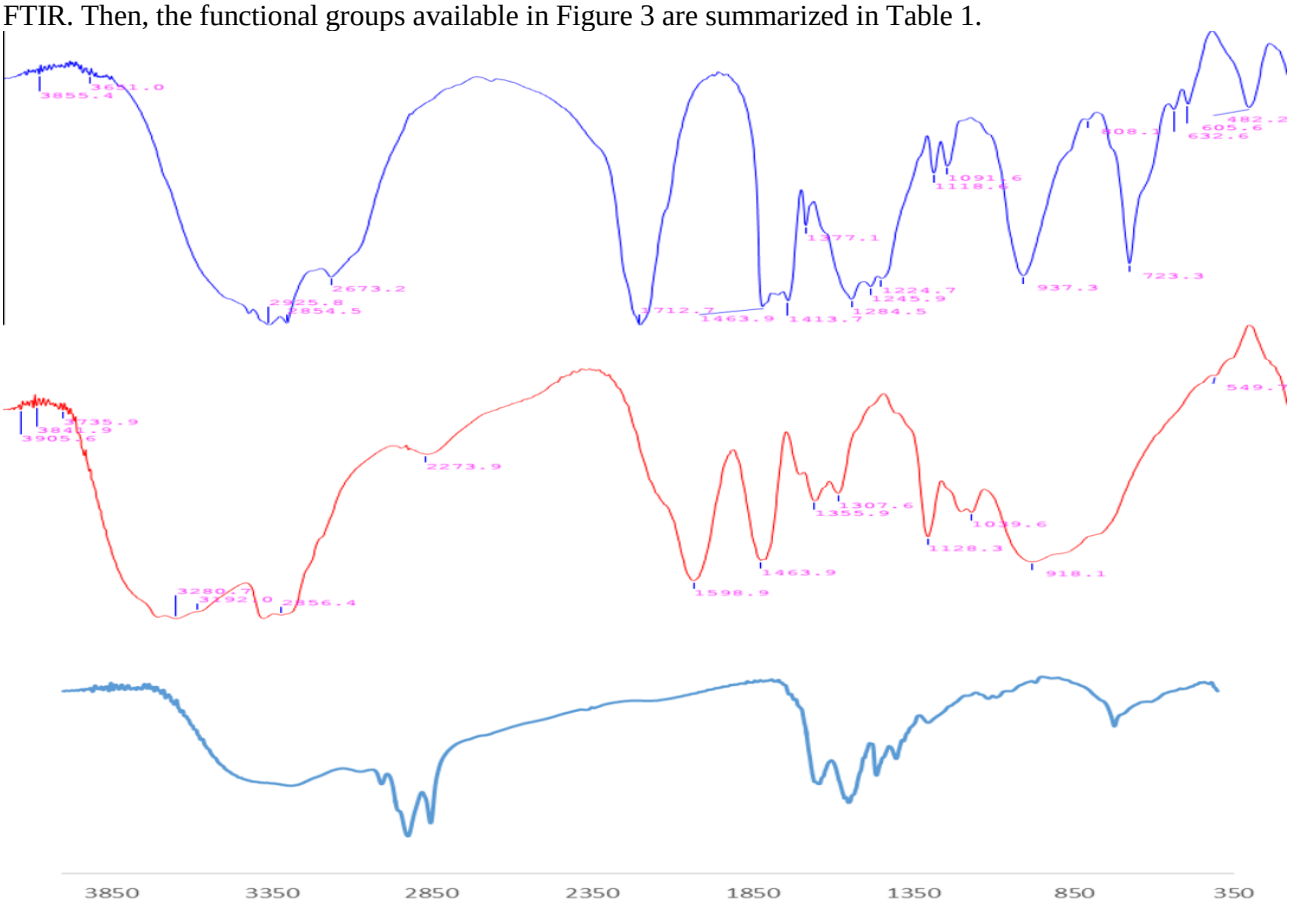

Figure 3. FTIR Spectrum of cis-oleic acid (above), DETA (middle), and cis-oleyl imidazoline (bottom)

Table 1. Functional Group Absorption in Cis-Oleyl Imidazolinium Iodide FTIR Spectrum

Functional Group	Wavenumber (cm^{-1})			
	cis-Oleat Acid	Dietilentriamin	cis-Oleyl Imidazoline	cis-oleyl imidazolinium iodida
O-H	3000-3500	-	-	-
C-H Stretch	2800-2900	2928-2856	2923.9-2854.9	2924.09
N-H Primer	-	3280-3192	-	-
N-H Sekunder	-	-	3286.5	3448.72
C=O	1712	-	1634.3	1637.56
C-N	-	1307.6	1303.8	1546.91
C=N	-	-	1552.6	1546.91
Skeletal Fatty	-	-	723.31	723.31

Then, Figure 4 shows the comparison of the FTIR spectra between cis-oleyl imidazoline and cis-oleyl-imidazolinium iodide. The spectrum in Figure 4 shows the absorption of a secondary N-H group (-NH-) at 3448.72 cm^{-1} which indicates the formation of cis-oleyl-imidazoline because NH_2 in DETA has reacted with oleic acid to form –NH- groups. It can also be seen the peak of C=N at 1546.91 cm^{-1}. The appearance of absorption in the C=N group indicates that a cyclization process has occurred during the reaction of DETA with oleic acid to become cis-oleyl-imidazoline.

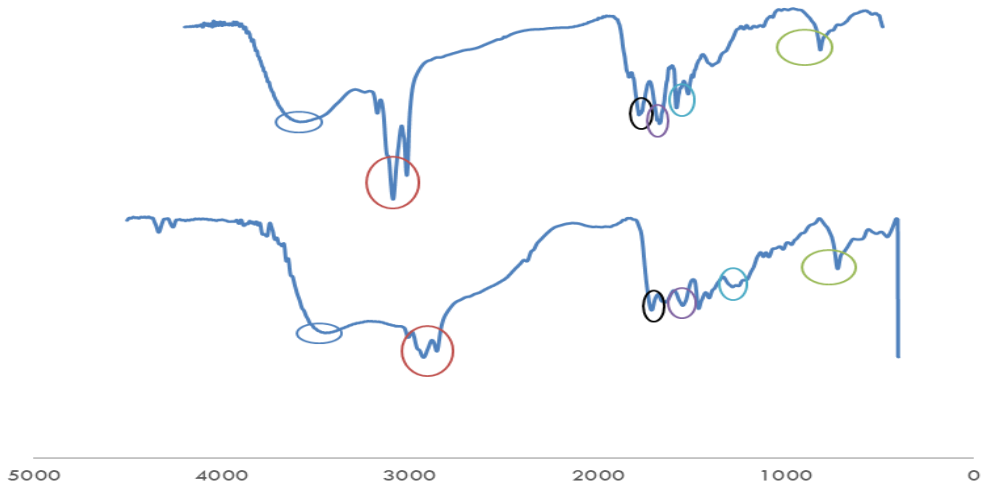

Figure 4. FTIR spectra of cis-oleyl imidazoline (top) and cis-oleyl-imidazolinium iodide (bottom)

3.4. Chemical characterization of cis-oleyl-imidazolinium with ^{1}H NMR and ^{13}C NMR

Figure 5 shows the results of the ^{1}H NMR analysis of cis-oleyl imidazoline. In this spectrum, there is a peak similar to the existing literature indicating a cis-oleyl imidazoline compound [16]. For more detail, the peaks in the NMR spectra are presented in Table 2.

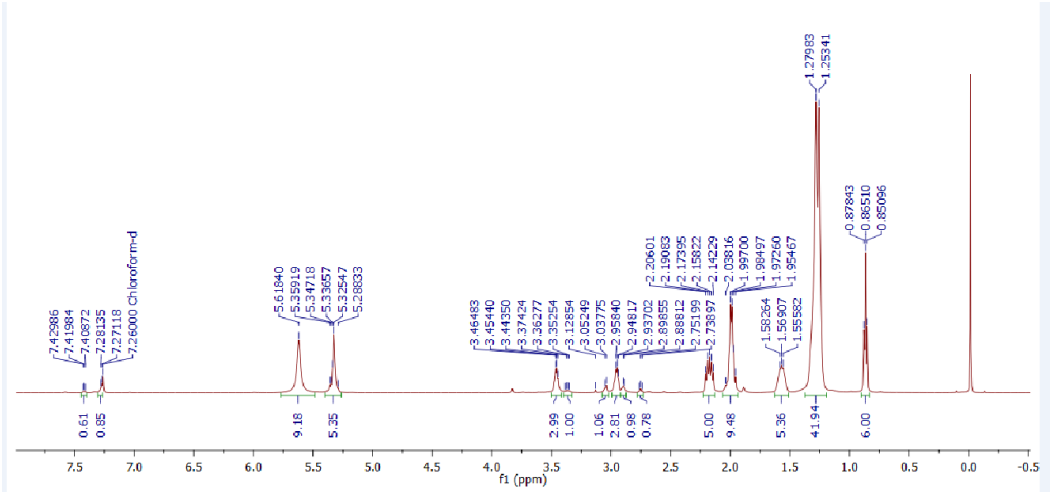

Figure 5. [1]H NMR spectrum of cis-oleyl Imidazolina

Table 2. Peak [1]H NMR Spectrum of cis-oleyl imidazolin

Peak (ppm)	Functional Group
0.86	CH_3
1.25-1.27	CH_2
1.55-1.58	CH_2—C amida
1.98-1.99	CH_2—C amida
2.14-2.20	CH_2—C imidazol
3.4	4H (2 CH_2 cincin imidazol)
5.28	CH (C=C *cis oleyl*)
5.6	CH
7.4	—CONH

Figure 6 and Table 3 show the peaks of the [13]C NMR spectrum of the cis-oleyl imidazoline compound. Then, Table 3 shows the [13]C NMR spectrum of the cis-oleyl imidazoline compound in detail. From the resulting spectrum, some peaks are similar to the existing literature and indicate that cis-oleyl imidazoline has been successfully synthesized [16].

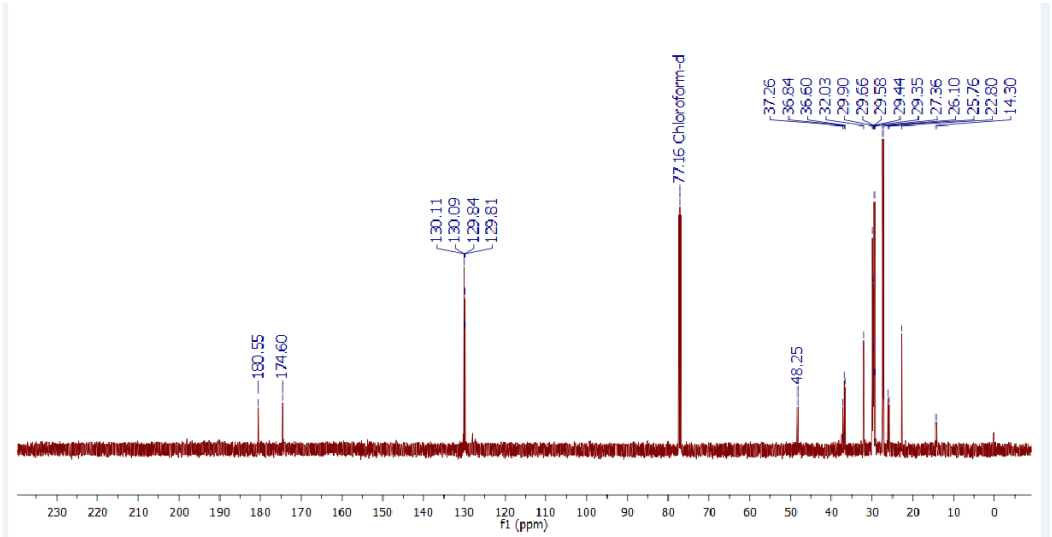

Figure 6. [13]C NMR spectrum of cis-oleyl imidazoline

Tabel 3. Spectrum of ^{13}C NMR cis-oleyl imidazoline

Peak (ppm)	Functional Group
14.30	CH_2—CH3
22.80	CH_2
29.33-29.66	CH_2 (cincin imidazol)
29.90	CN
32.03	CH_2—C (cincin imidazol)
36-37	CH_2—C (amida)
48	CH_2—N (amida)
129-130	CH (C=C *cis oleyl*)
174-175	R—C(=O)NR_2

Based on the results of the analysis, the synthesis reaction of cis-oleyl imidazoline is shown in Figure 7.

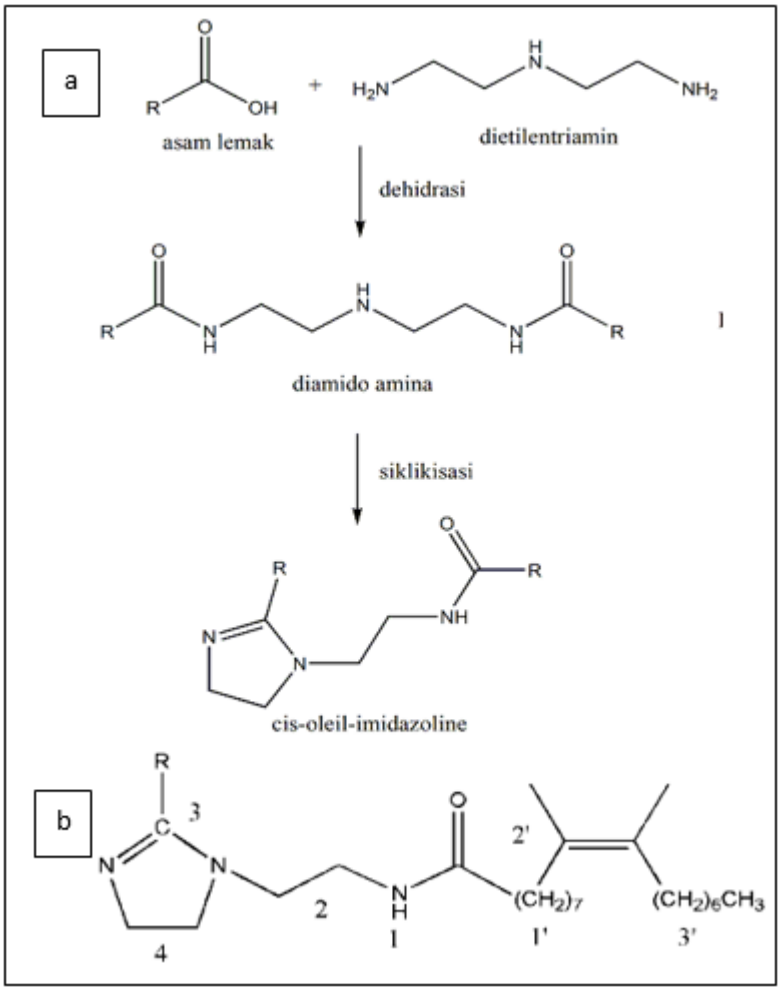

Figure 7. (a) reaction and (b) imidazoline cis-oleyl structure*

3.5. Chemical Characterization of Cis-Oleyl-Imidazolinium Iodide with ^{1}H NMR and ^{13}C NMR

Figures 8 and 9 are the results of ^{1}H and ^{13}C NMR. Interpretation of NMR is shown in the literature [18].

Figure 8 shows the peaks of the ^{1}H NMR spectrum from the synthesis step of the methylation-quaternary reaction producing a cis-oleyl imidazolinium iodide product. Peaks at 0.87; 1.26; 1.60-1.63; 2.0; 2.2-2.3; 3.33; 3.6-3.77; 5.33; and 7.61. From the resulting spectrum, some peaks are similar to the existing literature [15]. The peaks read are groups (CH_3), (CH_2)n, (CH_2 bound to C amide), (CH_2 bound to N amide), (CH_2 bound to C imidazole),(CH_2I bound to N imidazole), (CH_2 in the imidazole ring), (CH in -C=C- cis-oleyl), and (CONH-).

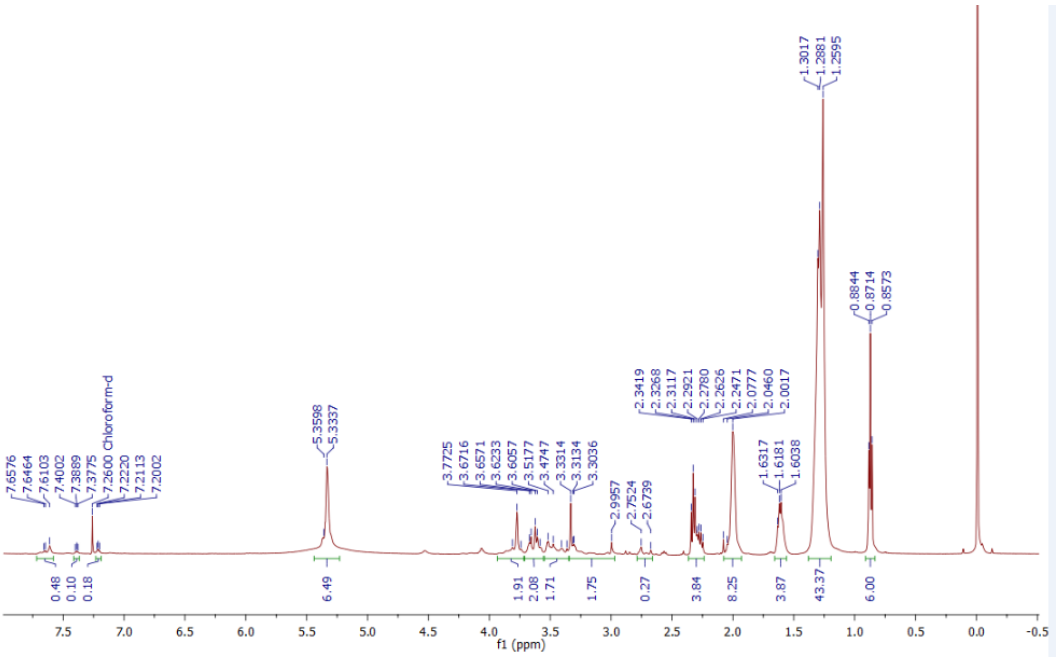

Figure 8. ^{1}H NMR spectrum of cis-oleyl Imidazoline

Figure 9 shows the peaks of the ^{13}C NMR spectrum. Peaks at, 14.2, 22.8, 24.89, 25.62, 27.29-27.35, 29.2-29.3, 29.45, 29.65, 29.8-29.9, 32.04, 34.17, 129.81-130.2 and 179.04. The peaks read are groups (CH_2-CH_3), (CH_2), (CH_2 in the imidazole ring), (CN), (CH_2 bound to the N imidazole), (CH_2-C in the imidazole ring), (CH_2-C in the amide), (CH_2-N in amides), (CH in -C=C-cis oleyl), and (R-C(=O)NR_2) were detected. From the resulting spectrum, some peaks are similar to the existing literature [16]. The difference between cis-oleyl imidazolinium iodide and the previous step, namely cis-oleyl imidazoline, is the appearance of a peak in the absorption range of 3.3 at ^{1}H NMR which is a CH_2I group and 29.45 absorptions at ^{13}C NMR, which is a CH_2 group bound to N on the imidazole ring, indicating that a methylation quaternization reaction of the synthesis of cis-oleyl imidazolinium iodide has been successful.

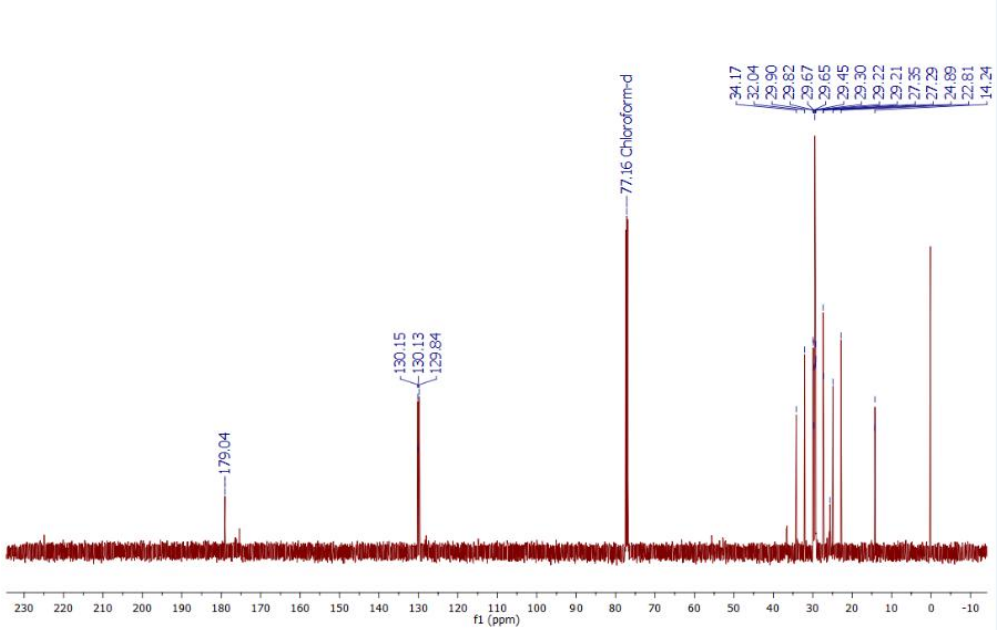

Figure 9. ^{13}C NMR spectrum of cis-oleyl imidazolinium iodide

Based on the characterization by FTIR and NMR, the mechanism of the methylation-quaternary reaction can be seen in Figure 10.

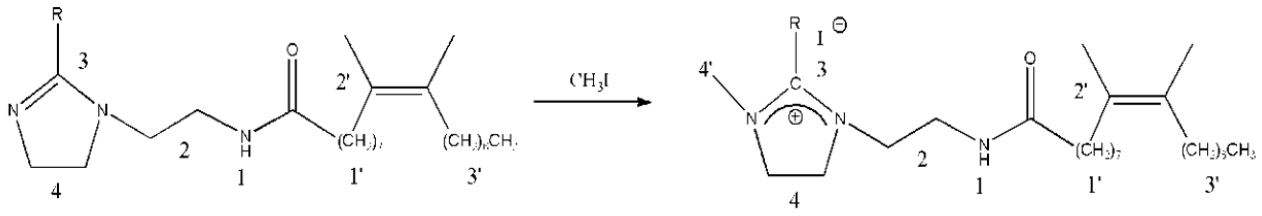

Figure 10. Quaternary methylation reaction of cis-oleyl imidazoline

3.6. Electrostatic activity test result

The electrostatic activity test on ceramic and floor coatings was carried out to determine the electrostatic activity of cis-oleyl-imidazolinium iodide added to the coating agent used. The activity test is in the form of measuring surface resistance including point-to-point resistance and ground resistance, as well as dust adhesion.

3.6.1. Test results for cis-oleyl-imidazolinium iodide and coating materials

The coating material used is polyurethane, which is known to have excellent friction resistance, wear resistance, and be quite stable to temperature changes. Cis-oleyl-imidazolinium iodide was mixed with a variation of 1-9 wt% Figure 11 shows the value of point-to-point resistance on polyurethane coating materials.

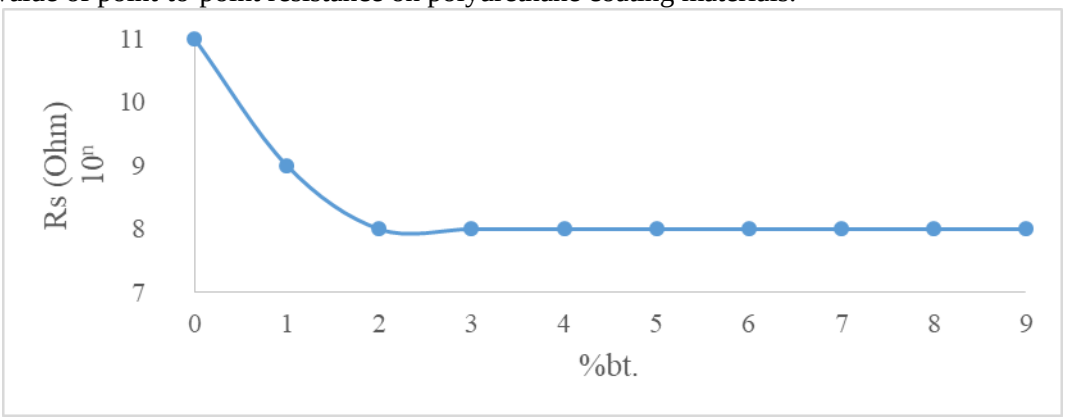

Figure 11. Point to point resistance in polyurethane coating materials

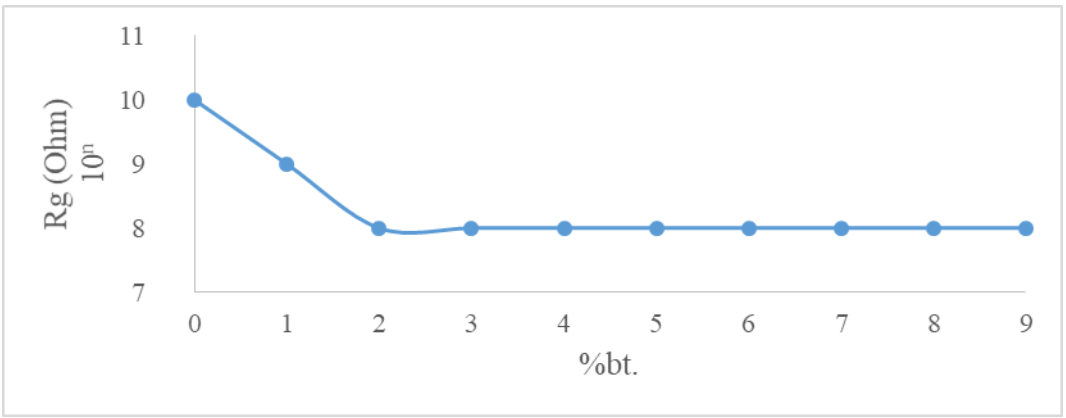

Figure 12. Ground resistance of mixed polyurethane coating material with cis-imidazolinium iodide

In Figure 11, it can be seen that the point to point Resistance (R_{PP}) on polyurethane is 1011 Ω which, although it is in the dissipative static range, is not safe enough because it is not in the sweet spot range of 105 – 10 9 Ω, and still tends

to be insulative. The coating mixture with IL shows a value of 109 for 9 wt%. and 108 for 2-9 wt%. cis-oleyl-imidazolinium iodide. The value of R_{pp} in the mixture shows that the coating properties are stative dissipative because they are in the range of 106 – 109 Ω. Likewise, the results of the ground resistance test (R_g) can be seen in Figure 12. That way, the coating can function properly because it will drain the electric charge slowly and safely to the ground.

3.6.2. Ceramic coating test results

Point to point and ground resistance tests were carried out on ceramics that had been coated with the addition of cis-oleyl-imidazolinium with variations of 0-9 wt% The result of the addition of IL causes a color change in the transparent coating to yellow according to the color of cis-oleyl-imidazolinium. This is a drawback because the surface color will not be as desired. Figure 13 is the physical appearance of the ceramic that has been coated.

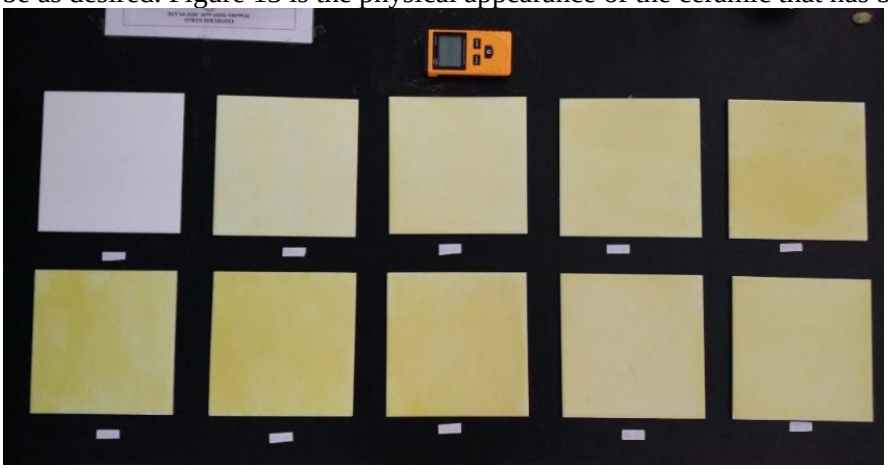

Figure 13. Result of ceramic coating with cis-oleyl-imidazolinium iodide

The results of the point-to-point resistance test and ceramic ground resistance can be seen in Figure 14 and Figure 15, respectively. Ceramic coating without the addition of cis-oleyl imidazolinium iodide showed R_{pp} and R_g values of 1012 Ω. This indicates that the ceramic is insulative and will not allow electrostatic current to flow. The electric charge will be stored for a long time and will continue to accumulate. This becomes dangerous because there can be a charged shock that causes high voltage electricity to fire.

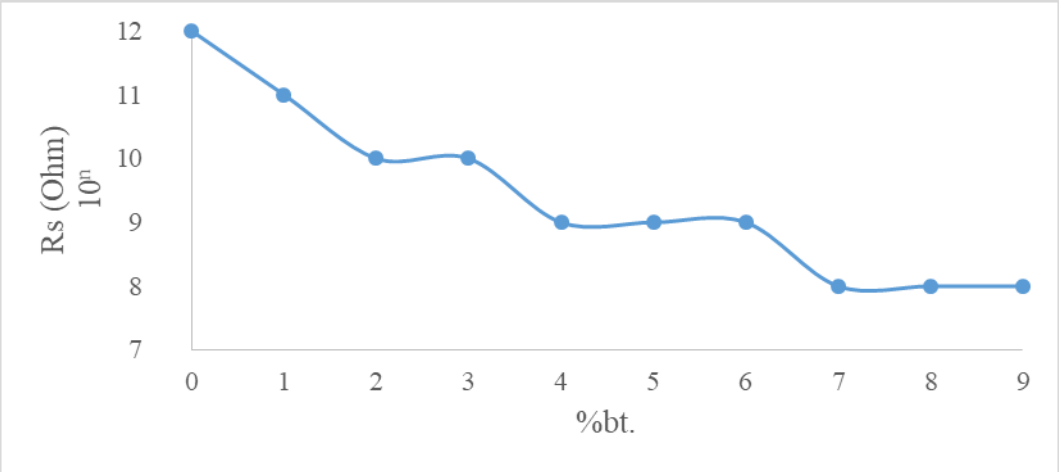

Figure 14. Point to point resistance of ceramics with cis-oleyl-imidazolinium iodide coating coating

In ceramic coatings with the addition of cis-oleyl-imidazolinium iodide, there are significant changes. The R_{pp} and R_g values are at 1011 Ω for 1 wt%., and 1010 Ω for 2-3 wt%. 109 Ω for R_g at 4-9 wt%., 109 Ω for R_{pp} at 4-6 wt%., and

108 Ω at 7-9 wt%. Ceramics are in a dissipative static state, making them safe and protected from possible explosions of charge or rapid electric currents that occur on conductive surfaces.

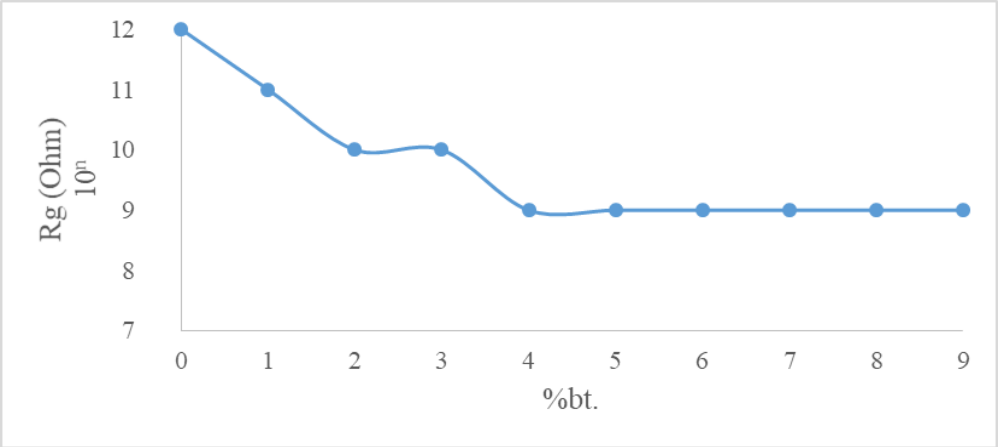

Figure 15. Ceramic ground resistance with cis-oleyl-imidazolinium iodide coating

3.6.3. Wood coating test results

Point-to-point resistance and ground resistance tests were carried out on wood that had been coated with the addition of cis-oleyl-imidazolinium with variations of 0-9 wt% The result of the addition of IL does not change the color of the wood surface. Figure 16 is the physical appearance of the wood after being coated.

Figure 16. Result of cis-oleyl-imidazolinium iodide coating on wood

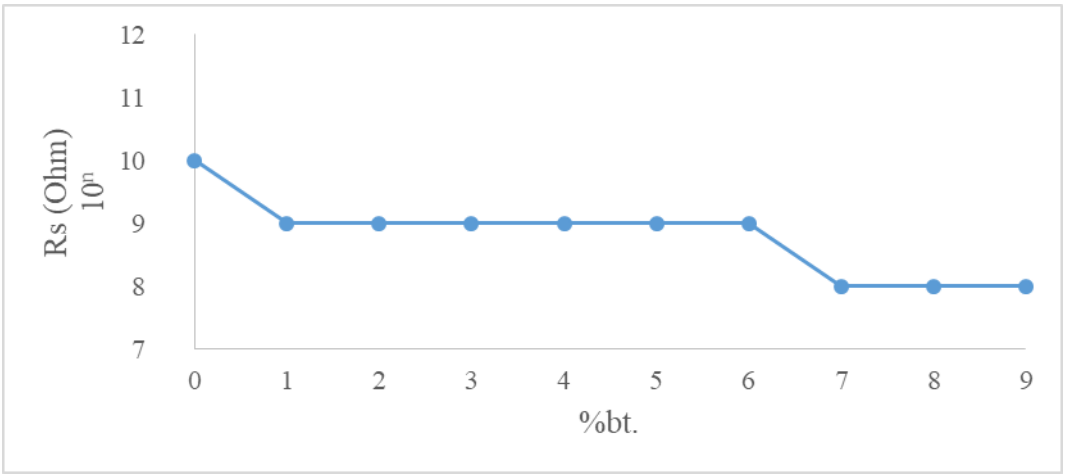

Figure 17. Point to point resistance of wood with cis-oleyl-imidazolinium iodide coating

The results of the resistance test can be seen in Figures 17 and 18. Wood coating without the addition of cis-oleyl Imidazolinium Iodide showed R_{pp} and R_g values of 1010 Ω. This indicates that the wood is statically dissipative, but

not yet in the sweet spot zone. In the wood coating with the addition of cis-oleyl-imidazolinium iodide, there were significant changes. R_{pp} value is at 109 Ω for 1-6 wt%., 108 Ω for 7-9 wt%. For R_g 1010 Ω at 1 wt%. and 109 Ω at 2-9 wt%. Wood is in a dissipative static state, making it safe and protected from possible explosions of charge or rapid electric currents that occur on conductive surfaces.

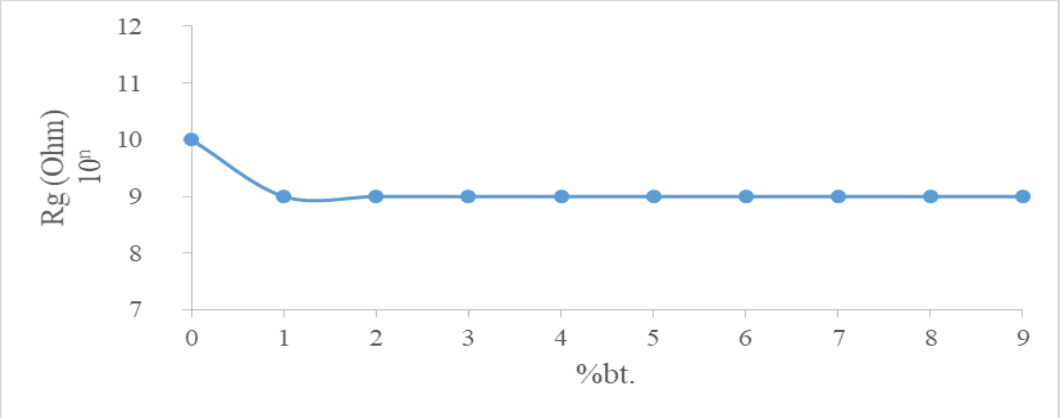

Figure 18. Ground resistance of wood with cis-oleyl-imidazolinium iodide coating

3.6.4. Dust adhesion test results

Figure 19 is an image of wood before (Figure 19(a)) and after (Figure 19(b)) coating for the dust adhesion test. In the dust adhesion test, activated carbon is used which is placed on the surface of ceramics and wood (see Figure 19(a)). The surface was previously rubbed with a cotton cloth 40 times to increase the charge on the surface. The wood and ceramic floors used are floors with an IL coating with a concentration of 7 wt%. The concentration was chosen because it is a concentration that does not affect the surface color but is already in the sweet spot zone.

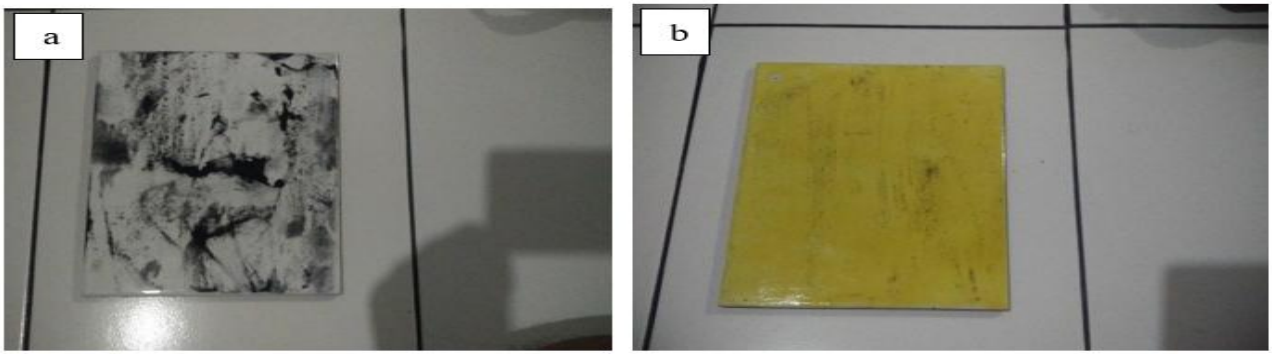

Figure 19. Ceramic floor dust adhesion test results without ILs (left) and with ILs (right)

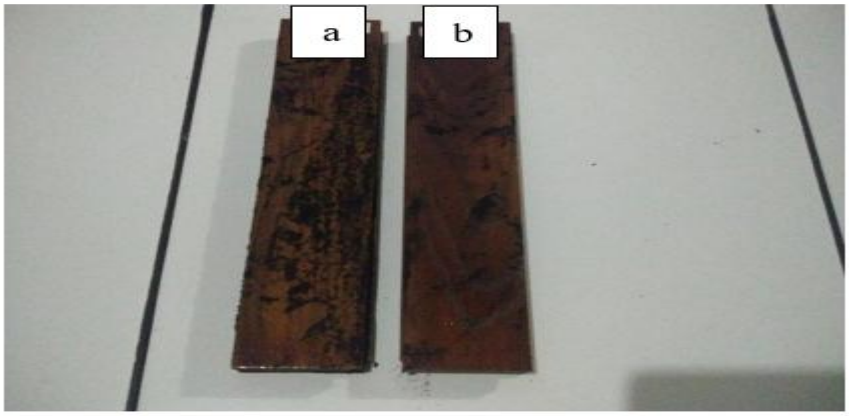

Figure 20. Wood floor dust adhesion test results without ILs (bottom) and with ILs (top)

The dust adhesion test results are shown in Figure 20. Based on Figure 20, the ceramic with the IL attracts much less activated carbon (see Figure 20(b)) than the ceramic without the IL (see Figure 20(a)). Likewise in Figure 20(b), the wood surface with the IL attracts much less activated carbon than the wood without the IL.

4. Conclusion

Synthesis of cis-oleyl imidazolinium iodide IL was carried out by reacting diethylenetriamine and cis-oleic acid in a ratio of 1:2 through an irradiation process, followed by quaternization methylation using methyl iodide. The results of FTIR, ^{1}H NMR, and 13C NMR characterization showed that the synthesis of cis-oleyl imidazolinium iodide IL was successful. The electrostatic activity of floor coating mixed with cis-oleyl imidazolinium iodide IL is statically dissipative because it is in the range of 106 – 109. The optimum Point to Point resistance for coating is 108 (2-9 wt%), ceramic coating is 108 (7-9 wt%), and wood coating is 108 (7-9 wt%). While the optimum Ground Resistance for coating is 108 (2-9 wt%), ceramic coating is 109 (4-9 wt%), and wood coating is 109 (1-9 wt%). Dust adhesion test results show that the mixed-coated floor attracts much less dust than the non-IL coating.

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